Final Report: Nanoscale Cluster Assembly on Compliant Substrates: A New Route to Epitaxy and Nanosctructure Synthesis

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From our work at the University of Minnesota prior to 2000, we knew that buffer-layer-assisted growth could be used to produce abrupt interfaces where reactions were constrained by the fact that particles derived from tens to millions of atoms were brought into contact with substrates that ranged from GaAs(110) to BiSrCaCuO superconductors [1, 10-19]. *In situ* scanning tunneling microscopy had demonstrated that the particles increased in size with the thickness of the buffer layer [9], and we postulated that buffer desorption somehow 'tossed the particles around' so that aggregation was possible. Through access to transmission electron microscopy in the MRL at the University of Illinois, we have been able to determine the distribution of particles delivered to amorphous carbon as a function of buffer thickness, buffer material, particle material, and warm up rate so as to reveal the physics underlying diffusion, aggregation, and coalescence. Significantly, this enhanced understanding makes it possible to design experiments that produce sizes and distributions of nanoparticles of a very wide range of materials.

The first experiments were designed to determine the particle densities and shapes for comparison to Monte Carlo simulations of cluster aggregation. Graduate student Christina Haley studied Au particles on amorphous carbon that had been formed by deposition onto Xe buffers of variable thickness [2]. She compared the fractal dimension for ramified particles to simulations and determined that it was consistent with 2D diffusion-limited cluster-cluster aggregation. She also showed that both the weighted average cluster size and the cluster density had a power law dependence on the buffer thickness.

When dealing with particles of nanometer dimension, it is important to realize that coalescence is driven by the very large surface energy and that particle integrity is a critical issue. We reasoned that the tendency of particles to coalesce could be altered by adsorbates that would reduce the surface energy. While the coalescence/sintering of metal particles of nanometer size had been modeled with molecular dynamic simulations and continuum models [20], clean experiments were few. Graduate student Vassil Antonov [3] investigated the coalescence of Pd nanostructures produced by BLAG under normal conditions and after coating them with an adsorbed monolayer of CO. The studies of Pd showed a behavior similar to Au, with a power law dependence of particle size on buffer thickness. He then repeated the experiments with a CO coating on the particles. He found that the CO layer was not sufficient to prevent aggregation, presumably because of irregularities in the particle shape. However, coalescence was significantly impeded, giving branched islands with thinner branches. The crossover size from compact to branched growth was reduced two-fold, indicating constraints in Pd atom diffusion related to the adsorbed CO. Another important conclusion was that solid CO performed the same function as Xe buffers allowing nanoparticle nucleation and growth, but with differences that reflected adatom and cluster interactions with the CO.

In BLAG, the initial nucleation of the clusters and their subsequent diffusion during warm-up should depend upon the buffer layer species. To quantify this dependence, graduate student Vassil Antonov, who was supported by DOE, worked with Jacob Palmer and Fulbright Fellow Arshad Bhatti to quantify the cluster density and the size distribution for Au particles as a function of buffer thickness for Xe, Kr, and Ar buffers [4]. For compact particles with a mean radius of less than ~5 nm radius, the diffusivity varied strongly with size and even increased with average size for a limited range. We attributed this increase in the apparent diffusivity to self-heating during coalescence (complete coalescence of two Au particles of 2000 atoms releases ~120 eV). Self-heating was more important for Kr than for Xe because of weaker interface

coupling. In the large-size limit, where the mean radius exceeded ~10 nm and the particles were branched (more than $2x10^5$ atoms), the diffusivity scaled with the inverse of the contact area with the buffer surface, consistent with MD simulations of fast slip diffusion on incommensurate surfaces. This suggests that the diffusivity is controlled by viscous friction between buffer and particle, an interesting example of friction at the nanoscale.

In BLAG, the temperature window in which desorption occurs is important in two competing processes. Since cluster diffusion is thermally activated, increasing the temperature increases the diffusivity. On the other hand, buffer desorption is also thermally activated, and increasing the temperature reduces the amount of time available for diffusion. Using the observed particle densities for different buffers, we were able to propose a model that quantified the competition between the processes. This also led us to look for buffers that might be effective in BLAG but which would desorb at significantly higher temperatures. For CO_2 buffers, the expected increase in diffusivity was offset by an increased barrier for diffusion due to the stronger interaction with the buffer [7]. Studies that are currently underway focus on H_2O [ice] as a buffer, and all indications are that it functions well.

In BLAG, one of the parameters that can be controlled is the rate at which the buffer layer is desorbed. Detailed investigations of the effect of buffer thickness and desorption rate on the extent of aggregation for metal particles on Xe [5], together with Monte Carlo simulations of cluster-cluster aggregation, made it possible to deduce the Arrhenius parameters for slip-diffusion. The effective activation energies ranged from 0.12 eV for small Ag clusters (a few hundred atoms) to 0.60 eV for ramified Ni islands (millions of atoms), and the pre-exponential factors varied over many orders of magnitude. Analyses of the barrier and the prefactors showed a correlation of the sort that has been observed in a wide range of physical, chemical, and biological processes, namely that an increase in the barrier is accompanied by a compensating exponential increase in the prefactor in a system of like processes. This Meyer-Neldel compensation law gave a characteristic energy of 6.9 meV, and we concluded that the optical phonons of solid Xe are responsible for nanostructure mobility. Moreover, we suggested that this dependence should be a common for nanoparticle diffusion. Studies by Philip Waggoner of diffusion on CO_2 buffers did, indeed, show an increase in the characteristic energy of the elementary excitations, consistent with the more energetic phonons of CO_2 [7]. Studies of H_2O buffers and C_{60} buffers should provide important insights, as well as determining the viability of BLAG at higher temperature.

Graduate student Jacob Palmer [8] undertook studies to determine whether the microstructure of the buffer layers used in BLAG could be used to vary the spatial distributions of the particles. First, we showed that the nuclei of rare gas layers on amorphous carbon or silica were very small, with irregular coverage at the beginning at 15 to 40 K. However, as the buffer grew in thickness, large columnar grains developed through secondary grain growth, analogous to what occurs in metal films at high temperature. Moreover, surface grooves were observed to form where the grain boundaries intersect the buffer surface, also in analogy to metal films at high temperature. The deposition of Au atoms onto these surfaces then produced clusters, and warm-up induced cluster diffusion and aggregation. What was new was that TEM results showed that the grooves were able to capture the mobile clusters in a fashion analogous to the capture of agitated grains of sand on a patterned surface. The grain boundaries and their grooves then offer a way to vary the cluster distribution on the surface and to print by pattern transfer using nanoscale particles of a material of choice [8].

Finally, Antonov and Parasuraman Swaminathan [10] considered the assembly of nanostructures of CdS, CdSe, and CdTe using the techniques of BLAG. He found that both compact clusters and ramified wires could be synthesized by varying the Xe buffer layer thickness. Analysis of the nanostructure size distributions and densities made it possible to calculate their diffusion parameters of the subliming Xe. Photoluminescence measurements indicated changes from 3D to 2D confinement as compact particles were replaced by ramified wires, and laser power dependent studies gave him the low temperature exciton lifetime.

A low temperature Omicron scanning tunneling microscope was acquired under DEFG02-01ER45944 to allow structural and electronic characterization of nanoparticles grown and maintained in ultrahigh vacuum, with control over temperature. Inaugural experiments were conducted by research associate Koji Nakayama that focused on Ag interactions with Br-passivated Si(100)-(2x1) [21]. Physical vapor deposition resulted in small 3D Ag structures, and subsequent annealing produced many-layer-high epitaxially-aligned Ag crystallites. We were able to obtain atomic resolution of the (111) top-most layer, demonstrate the absence of screw dislocations, and identify <-2 1 1> and <-1 1 0> steps that bounded the multilayer structure. This is the kind of information that will be needed to characterize the interactions of BLAG-delivered nanostructures.

In preparation for a new program that would deal with metal surfaces, we developed the protocol whereby Ag(111) films of high quality can be grown on Si(111)-(7x7), using the technique of deposition at low temperature and then crystallization at room temperature [22]. These surfaces are intended to be the substrates to which clusters can be delivered by BLAG.

Personnel:

When DEFG02-01ER45944 was funded, the PI had recently moved to Illinois. Only graduate student Christina Haley transferred with him, and she completed her M.S. in February 2002 [2]. Vassil Antonov joined in May 2001 and completed his PhD in December 2005. Jacob Palmer joined in September 2002 and Parasuraman Swaminathan joined in September 2003. Arshad Bhatti was a Fulbright Fellow in WeaverLabs from October 2002 – September 2003. After his return to Pakistan, we wrote successful proposals to the NSF-INT and the Pakistan Ministry of Science and Technology that will support personnel exchange and enhance the proposed continuation of DEFG02-01ER45944. Undergraduate Philip Waggoner worked on BLAG beginning in his sophomore year. In September 2005, he matriculated at Cornell in Applied Physics with an NDSEG fellowship. Postdoctoral research associate Koji Nakayama joined in March 2001 and was been supported in part by DEFG02-01ER45944; his focus was been on the development of low temperature STM, studies of the Ag cluster stability on Br-Si(100) [23], single atom chemical recognition [24], and preparation for BLAG growth in the STM. He took a permanent position at IMR at Sendai University in June 2005.

Refereed publications:

Christina Haley and J.H. Weaver, "Buffer-layer-assisted nanostructure growth via two-dimensional cluster-cluster aggregation," Surf. Sci. 518, 243-250 (2002).

V.N. Antonov and J.H. Weaver, "CO-induced morphology modification in buffer-layer-assisted growth of Pd nanostructures," Surf. Sci. 526, 97-106 (2003).

V.N. Antonov, J.S. Palmer, A.S. Bhatti, and J.H. Weaver, "Nanostructure diffusion and aggregation on desorbing rare-gas solids: Slip on an incommensurate lattice," Phys. Rev. B 68, 205148 (2003).

V.N. Antonov, J.S. Palmer, P.S. Waggoner, A.S. Bhatti, and J.H. Weaver, "Nanoparticle diffusion on desorbing solids: The role of elementary excitations in buffer-layer-assisted growth," Phys. Rev. B 70, 045406 (2004).

J.H. Weaver and V.N. Antonov, "Synthesis and patterning of nanostructures of (almost) anything on anything," Surf. Sci. 557, 1-3 (2004). See also C&E News **82**, 6 (May 3, 2004), Science Editor's Choice **304**, 797 (2004), and Physics Today, pp. 22-23, June 2004.

J.S. Palmer, V.N. Antonov, A.S. Bhatti, P. Swaminathan, P.S. Waggoner, and J.H. Weaver, "The effects of buffer structure on buffer-layer-assisted growth: Grain boundaries, grooves, and pattern transfer," Surf. Sci. 595, 64-72 (2005).

P.S. Waggoner, J.S. Palmer, V.N. Antonov, and J.H. Weaver, “Metal nanostructure growth on buffer layers of molecular CO_2,” Surf. Sci. 596, 12-20 (2005).

K.S. Nakayama and J.H. Weaver, “Formation and stability of Ag mounds on Br-Si(100) and their role in pit formation,” Surf. Sci. 574, 331-337 (2005).

K.S. Nakayama, T. Sugano, K. Ohmori, and J.H. Weaver, “Chemical fingerprinting at the atomic limit with scanning tunneling spectroscopy,” Surf. Sci. 600, 716-723 (2006).

V.N. Antonov, P. Swaminathan, J.A.N.T. Soares, J.S. Palmer, and J.H. Weaver, “Photoluminescence of CdSe quantum dots and rods from buffer-layer-assisted growth,” Appl. Phys. Lett. 88, 121906 (2006).

P. Swaminathan, V.N. Antonov, J.A.N.T. Soares, J.S. Palmer, and J.H. Weaver, “Cd-based II-VI semiconductor nanostructures produced by buller-layer-assisted growth: Structural evolution and photoluminescence,” Phys. Rev. B 73, 125430 (2006).

Invited presentations:

J.H. Weaver, "Nanostructure Formation, Integration, and Stability," European Conference on Surface Science-20, Krakow, Poland, September 2002

J.H. Weaver, "Nanoscale Engineering," 22nd Werner Brandt Workshop, Namur, Belgium, June 2002.

J.H. Weaver, "Science at the Nanometer Scale," Plenary talk at the Mexican Vacuum Society, Veracruz, Mexico, October 2002. [Unable to attend.]

J.H. Weaver, "Three Dimensional Nanoengineered Assemblies," Materials Research Society Meeting, Boston, November 2002.

J.H. Weaver, "Three Dimensional Nanoengineered Assemblies," Kodak Distinguished Lecture in Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, February 2003.

J.H. Weaver, “Formation, Characterization, and Integration of Nanostructures,” Colloquium, Northwestern University, Evanston, February 2003.

J.H. Weaver, "Buffer-Layer-Assisted Growth and the Formation of Nanostructures of (Almost) Anything on Anything," AVS Distinguished Lecture, Michigan Chapter, Ann Arbor, May 2003.

J.H. Weaver, "Nanoscale Surface Engineering," Thirteenth International School on Vacuum, Electron, and Ion Technologies, Varna, Bulgaria, September 2003.

J.H. Weaver, "Buffer-Layer-Assisted Growth and the Formation of Nanostructures of (Almost) Anything on Anything," AVS Distinguished Lecture, New Mexico Chapter, Albuquerque, May 2004.

J.H. Weaver, V.N. Antonov, J.S. Palmer, and A.S. Bhatti, “Nanoparticle Diffusion and Aggregation on Desorbing Rare Gas Solids: Slip on an Incommensurate Lattice,” IVC-16/ICSS-12/NANO-8, Venice, June 2004.

J.H. Weaver, “Buffer-Layer-Assisted Growth and the Formation of Nanostructures of (Almost) Anything on Anything,” International Conference on Surface and Interface Science, Tel Aviv, October 2004.

J.H. Weaver, “Nanoscience and the Synthesis and Characterization of Nanometer Structures,” Plenary Lecturer, Pakistan Physical Society Meeting, Lahore, January 2005.

J.H. Weaver, five lectures on nanoscience and technology, Islamabad, January 2005.

J.H. Weaver, Nanoscience and the Synthesis and Characterization of Nanometer Structures," Ghulam Ishaq Khan Institute, Topi, January 2005.

J.H. Weaver, three lectures on nanoscience and surface patterning, 30th International Nathiagali Summer College, Islamabad, July 2005.

J.H. Weaver, "Synthesis and Characterization of Metallic and Semiconductor Nanoparticles on Surfaces," AVS Prairie Chapter Meeting, Naperville, June 2006.

Contributed presentations:

V.N. Antonov, C.L. Haley, and J.H. Weaver, "Buffer-Layer-Assisted Nanostructure Growth via Two-Dimensional Cluster-Cluster Aggregation," 50th Midwest Solid State Conference, Urbana, October 2002.

C.L. Haley, V.N. Antonov, and J.H. Weaver, "Buffer-Layer-Assisted Nanostructure Growth via Two-Dimensional Cluster-Cluster Aggregation," 49th International Symposium of the AVS, Denver, November 2002.

A.S. Bhatti, V.N. Antonov, J.S. Palmer, and J.H. Weaver, "On the Growth Kinetics of Nanostructures on a Desorbing Buffer Layer," Nanotechnology Workshop, Urbana, May 2003.

V.N. Antonov, J.S. Palmer, A.S. Bhatti, and J.H. Weaver, "Diffusion-Limited Cluster-Cluster Aggregation on Surfaces of Desorbing Rare Gas Solids," Understanding Complex Systems Symposium, Urbana, May 2003.

V.N. Antonov, J.S. Palmer, A. Bhatti, and J.H. Weaver, " Influence of Buffer Composition on Buffer-Layer-Assisted Growth and Absorption Spectroscopy on Au Nanostructures," 25th Annual Symposium on Applied Surface Science, Urbana, June 2003.

V.N. Antonov, J.S. Palmer, A.S. Bhatti, and J.H. Weaver, "Mobility of Nanostructures on the Surface of a Desorbing Solid: Friction at the Nanoscale," 50th International Symposium of the AVS, Baltimore, November 2003.

V.N. Antonov, J.S. Palmer, P.S. Waggoner, A.S. Bhatti, and J.H. Weaver, "Nanoparticle Diffusion on Desorbing Solids: The Role of Elementary Excitations in Buffer-Layer-Assisted Growth," Understanding Complex Systems Symposium, Urbana, May 2004. Antonov won "best poster of the conference."

V.N. Antonov, J.S. Palmer, P.S. Waggoner, A.S. Bhatti, and J.H. Weaver, "Nanoparticle Diffusion on Desorbing Solids: The Role of Elementary Excitations in Buffer-Layer-Assisted Growth," AVS Prairie Chapter, Urbana, June 2004.

V.N. Antonov, J.S. Palmer, P.S. Waggoner, A.S. Bhatti, and J.H. Weaver, "Nanoparticle Diffusion on Desorbing Solids: The Role of Elementary Excitations in Buffer-Layer-Assisted Growth," AVS 51st International Symposium, Anaheim, November 2004. Antonov won the top student award of the Society, the Russell and Sigurd Varian Award.

P.S. Waggoner, J.S. Palmer, V.N. Antonov, and J.H. Weaver, "Metal Nanostructure Growth on Buffer Layers of Molecular CO_2," CNST Workshop on Nanotechnology, Urbana, May 2005.

V.N. Antonov and J.H. Weaver, "Kinetics of Diffusion and Self-Assembly of Metal and Semiconductor Nanostructures on Rare Gas Solids," 65th Annual Physical Electronics Conference, Madison, June 2005. Antonov competed for the Nottingham Prize.

J.S. Palmer, V.N. Palmer, A.S. Bhatti, P.S. Waggoner, P. Swaminathan, and J.H. Weaver, "Rare Gas Solid Grains and Grooves: The Influence of Film Structure on Buffer-Layer-Assisted Nanostructure Assembly," 65th Annual Physical Electronics Conference, Madison, June 2005.

P.S. Waggoner, J.S. Palmer, V.N. Antonov, and J.H. Weaver, "Buffer-Layer-Assisted Growth on Molecular Buffers: Metallic Nanostructures on CO_2," 65th Annual Physical Electronics Conference, Madison, June 2005.

J.S. Palmer, V.N. Antonov, A.S. Bhatti, P. Swaminathan, P.S. Waggoner, and J.H. Weaver, "The Effects of Buffer Structure on Buffer-Layer-Assisted Growth: Grain Boundaries, Grooves, and Pattern Transfer," Dynamics of Materials Revealed by Electron Microscopy, Urbana, June 2005.

V.N. Antonov, J.S. Palmer, A.S. Bhatti, and J.H. Weaver, "Buffer-Layer-Assisted Growth and the Formation of Nanostructures of (Almost) Anything on Anything," Dynamics of Materials Revealed by Electron Microscopy, Urbana, June 2005.

V.N. Antonov, J.S. Palmer, A.S. Bhatti, and J.H. Weaver, "Buffer-Layer-Assisted Growth and the Formation of Nanostructures of (Almost) Anything on Anything," 30th International Nathiagali Summer College on Physics and Contemporary Needs, Islamabad, Pakistan, July 2005.

J.S. Palmer, V.N. Antonov, A.S. Bhatti, P. Swaminathan, P.S. Waggoner, and J.H. Weaver, "Rare Gas Solid Grains and Grooves: The Influence of Film Structure on Buffer-Layer-Assisted Nanostructure Assembly," 30th International Nathiagali Summer College on Physics and Contemporary Needs, Islamabad, Pakistan, July 2005.

A.S. Bhatti, P. Swaminathan, V.N. Antonov, J.S. Palmer, and J.H. Weaver, "Photoluminescence from a-Ge Nanoparticles: Confinement Effects," 30th International Nathiagali Summer College on Physics and Contemporary Needs, Islamabad, Pakistan, July 2005.

P.S. Waggoner, J.S. Palmer, V.N. Antonov, and J.H. Weaver, "Buffer-Layer-Assisted Growth on Molecular Buffers: Metallic Nanostructures on CO_2," 52nd International Symposium of the AVS, Boston, October 2005.

V.N. Antonov, P. Swaminathan, J.S. Palmer, and J.H. Weaver, "Self-Assembly and Photoluminescence of CdSe Quantum Dots from Buffer-Layer-Assisted Growth," 52nd International Symposium of the AVS, Boston, October 2005.

P. Swaminathan, V.N. Antonov, J.A.N.T. Soares, J.S. Palmer, A.S. Bhatti, and J.H. Weaver, "II-VI Semiconductor Nanostructures Produced by Assembly on Rare Gas Solids," CNST Nanotechnology Workshop, Urbana 2006.

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6. J.H. Weaver and V.N. Antonov, "Synthesis and Patterning of Nanostructures of (Almost) Anything on Anything," Surf. Sci. **557**, 1-3 (2004).
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22. I. Matsuda *et al.*, “Growth and Electron Quantization of Metastable Silver Films on Si(001),” Phys. Rev. B **63** 125325 (2001).
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